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(54) **Plastic closure with compression molded sealing liner**

Kunststoffverschluss mit formgepresstem Abdichtungsbelag

Fermeture en plastique avec revêtement d'étanchéité moulé par compression

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(56) References cited:  
**EP-A- 0 068 148 EP-A- 0 421 620  
EP-A- 0 521 426 CA-A- 2 025 442**

- **DATABASE WPI Week 9129, Derwent  
Publications Ltd., London, GB; AN 91-211817 &  
JP-A-3 134 085 (ASAHI CHEM. ET AL) 7 June  
1991**
- **DATABASE WPI Week 9248, Derwent  
Publications Ltd., London, GB; AN 92-395040 &  
JP-A-4 294 754 (ASAHI CHEM IND) 19 October  
1992**

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## Description

[0001] This invention relates to molded plastic closures and particularly to compression molded plastic closures having compression molded liners therein.

[0002] Plastic closures have been commonly made by injection molding or compression molding. In plastic closures made by compression molding as shown, for example, in U.S. Pat. Nos. 3,210,805, 4,343,754 and 4,418,828, it is also common to compression mold a sealing liner in place in the closure after the closure has been formed. Such sealing liners may comprise polyvinyl chloride plastic or thermoplastic elastomeric compositions. Polyvinyl chloride has a tendency to overheat during extrusion and further has inherent problems because of the removal torque when the sealing liner is used on a PET container. Thermoplastic polymeric compositions are most expensive and are more difficult to produce in that the compounding necessary results in inconsistent quality and consistency. Furthermore, such compositions produce problems of torque removal on glass containers. U.S. Pat. Nos. 4,807,772 and 4,845,362 disclose a compression molded liner being made of a blend of polyethylene or polypropylene and thermoplastic elastomeric copolymer so that the liner is removable.

[0003] As a result of the problems with both types of compositions, it has been necessary for a user to maintain costly inventories of closures with both types of sealing liners in order to accommodate use with both plastic and glass containers.

[0004] In order to produce a satisfactory sealing liner that would be applicable to both plastic and glass containers, it must be necessary to not only formulate the composition but also consistently extrude and cut off charges of the composition for compression molding in apparatus such as shown, for example, in U.S. Pat. Nos. 3,955,605, 4,080,136 and 4,277,431. The closure must also match other closure requirements including satisfactory removal torques, carbonation retention, and carbonation retention under load and satisfactory functioning on a container when cycled through environmental changes of temperature and humidity.

[0005] In U.S. Pat. No. 4,984,703 (corresponding to EP-A-0 421 620) there is disclosed a compression molded plastic closure having a compression molded sealing liner which fulfills the requirements of a carbonated beverage closure and operates satisfactorily on both plastic and glass containers.

[0006] When a plastic beverage cap has been provided with an ethylene vinyl acetate liner, it has been found that erratic high removal torques have been provided, the erratic high removal torques are not eliminated. Various efforts have been proposed as disclosed in U.S. Pat. Nos. 3,400,094, 3,406,854, and 4,463,116 that utilize lubricants but, as far as the present inventor knows, have been without success. Specification EP-A-0 521 426 describes compositions containing ethylene vinyl acetate and mixtures of fatty amides in the production of foils.

[0007] Among the objectives of the present invention are to provide a plastic closure with a sealing liner that comprises an ethylene vinyl acetate composition and includes other components that provide for uniform removal torques; wherein the closure is useful in cold climate conditions with migration of the lubricant to the surface of the liner; wherein the liner can be compression molded in the plastic closure; or where the liner can be molded and then inserted in the closure.

[0008] In accordance with the invention, a plastic closure includes a base wall having an inner surface, a peripheral skirt extending from the base wall and a liner on the inner surface of the base wall. The liner comprises (a) ethylene vinyl acetate composition, (b) a primary fatty acid amide and (c) a bis-fatty acid amide. The fatty acid amide comprises an oleamide or erucamide, and the bis-fatty acid amide preferably comprises ethylene-bis-stearamide.

[0009] The oleamide or erucamide and the bis-fatty acid amide are preferably mixed before introduction into the ethylene vinyl acetate composition. The total amount of fatty acid amide and bis-fatty acid amide to the ethylene vinyl acetate is 0.5% - 1.5% by weight.

[0010] The ratio of fatty acid amide to bis-fatty acid amide is preferably 50-50 by weight but may range between 25:75 and 75:25 by weight. The ethylene vinyl acetate composition comprises 85% - 94% by weight polyethylene and 6% - 15% by weight vinyl acetate.

[0011] The plastic closure embodying the invention is made of suitable material such as polypropylene and comprises a base wall, a peripheral skirt having internal threads for engagement with complementary threads on a container and a sealing liner in the form of an annular shape at the juncture of the base wall and peripheral skirt. Such a closure may be of the type disclosed in United States patent 4,984,703 (EP-A-0 421 620), incorporated herein by reference. The closure may include tamper indicating means such as shown, for example, in U.S. Pat. Nos. 4,418,828, 4,613,052 and 4,653,657.

[0012] In accordance with the invention, the sealing liner comprises an ethylene vinyl acetate composition and a blend of a primary acid amide and bis-fatty acid amide, which are believed to function as lubricants.

[0013] The oleamide or erucamide and the bis-fatty acid amide are preferably mixed before introduction into the ethylene vinyl acetate composition. The total amount of fatty acid amide and bis-fatty acid amide to the ethylene vinyl acetate is 0.5% - 1.5% by weight.

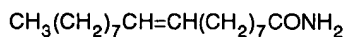
[0014] The ratio of fatty acid amide to bis-fatty acid amide is preferably 50-50 by weight but may range between 25:75 and 75:25 by weight.

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[0015] The ethylene vinyl acetate composition comprises polyethylene as the major constituent and vinyl acetate and optionally includes antioxidants and pigments.

[0016] Fatty acid amides are based on fatty saturated and unsaturated straight-chain, monobasic acids derived from naturally occurring feedstocks. Predominant chain lengths range from eighteen to twenty-two carbon atoms. The primary amides are waxy solids with melting points in the range of 68-108°C.

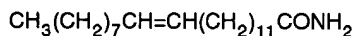
[0017] Oleamide fatty acid amides comprise an unsaturated fatty primary amide derived from oleic acid wherein the major component is:



[0018] Such an oleamide has the following physical properties:

TYPICAL PROPERTIES	KEMAMIDE O
Molecular weight	275
Density @ 130°C(266°F), g/mL	0.82
	2
Density @ 160°F(71°C),g/mL	0.85
	7
Viscosity @ 130°C,cP	5.5
Heat of fusion, kcal/mol	6.3
Flash point,°C,COC	245
Fire point,°C,COC	264

[0019] Erucamide fatty acid amides comprise unsaturated fatty primary amide derived from erucic acid wherein the major component is:

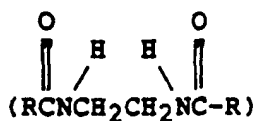


[0020] Such an erucamide has the following physical properties:

TYPICAL PROPERTIES	KEMAMIDE E
Molecular weight	335
Density @ 130°C(266°F),g/mL	0.81
	5
Density @ 160°F(70°C),g/mL	0.85
	1
Viscosity @ 130°C,cP	6.4
Heat of fusion,kcal/mol	9.8
Flash point,°C,COC	262
Fire point,°C,COC	287

[0021] Ethylene bisamides are reaction products of saturated or unsaturated fatty acids and ethylenediamine.

[0022] A satisfactory fatty bisamide derived from stearic acid is: N,N'-ethylenebisstearamide when the major component is:



where R = stearyl

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[0023] This fatty bisamide has the following properties:

TYPICAL PROPERTIES	KEMAMIDE W-40
Flash point, °C, COC	299
Fire point, °C, COC	315
Physical form	Flake Only

[0024] Another satisfactory fatty bisamide derived from oleic acid is N,N'-ethylenebisoleamide herein the major component is:



where R = oleyl

[0025] This fatty bisamide has the following properties:

TYPICAL PROPERTIES	KEMAMIDE W-20
Flash point, °C, COC	296
Fire point, °C, COC	315
Physical form	Powder and pellet

[0026] The ethylene vinyl acetate composition has a composition within the following ranges, by weight:

Polyethylene	85% to 94%
Vinyl acetate	6% to 15%

[0027] The primary acid amide comprises either oleamide or erucamide.

[0028] The bis-fatty acid amide preferably comprises an ethylene-bis-stearamide.

[0029] The liner composition may include antioxidants and colorants as is conventionally used.

[0030] In a preferred form, the primary acid amide and bis' fatty acid amide are mixed with a colorant and a carrier such as low density polyethylene and then this is provided for use with the ethylene vinyl acetate composition.

[0031] In an early experiment, the resins used were as follows:

RESIN	MELT FLOW (gm/10 min)	DENSITY (gm/cc)	ACETATE%
Dupont Elvax 750	7.0	0.930	9.0
Dupont Elvax 650	8.0	0.933	12.0
Dupont Elvax 550	8.0	0.935	15.0
Dupont Elvax 450	8.0	0.940	18.0

[0032] Elvax is a trademark of Dupont for ethylene vinyl acetate copolymer.

[0033] The various lubricants used in such experiments were as follows:

## LUBRICANTS

[0034]

- Kemamide U (oleamide)
- Kemamide E (erucamide)
- Kemamide W-40 (ethylene-bis-stearamide)
- Kemamide W-20 (ethylene-bis-oleamide)

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[0035] Kemamide is a trademark of Humko Chemical Division of Witco Corporation.

[0036] As used herein, the components of the composition are abbreviated as follows:

olea = oleamide

eruc = erucamide

EBS = ethylene bis stearamide

EBO = ethylene bis oleamide

[0037] In such an experiment testing for coefficient of friction (COF), mixtures of the additives intended to function as lubricants were provided as follows with the results indicated:

ratio	olea/eruc	EBS/EBO	EBS/eruc	EBS/olea	EBO/eruc	EBO/olea
100/0	1.040	2.970	2.970	2.970	4.050	4.050
50/50	0.900	2.790	1.060	0.990	1.280	1.10
0/100	1.510	3.970	1.510	1.040	1.510	1.040

[0038] With the exception of the EBO/oleamide mixture, the binary mixtures exhibit lower coefficient of friction (COF) values than the individuals by themselves. This approach was extended in the erucamide and oleamide systems mixed with EBS and EBO to find the optimum ratio of the lubes that would give the minimum COF.

[0039] The following tables show the result at 1% total concentration of lubricant with the ethylene vinyl acetate (EVA):

%EBS	EBS/Oleamide and Erucamide Mixes COF (erucamide mix) - (oleamide mix)	
0%	1.510	1.040
25%	1.340	1.030
50%	1.060	0.990
75%	1.270	1.330
100%	2.970	2.970

%EBO	EBO/Oleamide and erucamide Mixes COF (erucamide mix) - (oleamide mix)	
0%	1.510	1.040
25%	1.090	0.990
50%	1.330	1.110
75%	1.260	1.170
100%	3.870	3.870

[0040] The data shows that the 50:50 mix of EBS to erucamide and or oleamide produces the optimum ratio whereas with EBO mixtures, the optimum ratio is 25% EBO blended with erucamide or oleamide.

Concentration of Mixtures:

[0041] The effects of total concentration of the mixtures were explored. COF measurements of 50:50 EBS mixtures with erucamide and oleamide are shown in the following table:

% total lubricants	erucamide/EBS	oleamide/EBS
1.0%	1.060	0.990
0.75%	1.100	1.070
0.5%	1.110	1.120

[0042] As when erucamide and oleamide are utilized as single components, the concentration of the mixed lubricants only slightly affects the COF in the range of concentrations tested.

[0043] In further tests, the following results in removal torques were obtained:

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COMPOSITIONS					
	CONTROL	A	B	C	D
ELVAX 750	-	-	100	100	-
ELVAX 650	100	100	-	-	100
EBS	-	0.5	0.5	0.5	0.5
ERUCAMIDE	-	0.5	0.5	-	-
EBO	1.5	-	-	-	-
OLEAMIDE	-	-	-	0.5	0.5
LDPE (30MF)	10	10	10	10	10
PACK RESULTS:					
REMOVAL TORQUE (RT)	12.0	7.8	8.2	8.2	7.6
IMMEDIATE					
1 DAY	11	10	10.3	12.6	11.2
1 WEEK	15.0	13.8	14.0	12.6	14.0
CO2-1 WEEK	3.85	3.84	3.80	3.86	3.85

[0044] It can be seen that the compositions A, B, C, D provided lower removal torques than the EVA control alone.

[0045] Other tests covering immediate and removal torques, comprises the compositions A and C:

	A	Control
<b>Immediate</b>		
avg.	14.2	19.4
sigma	1.27	1.51
mn/mx	12/16	16/22
<b>1 Day</b>		
avg.	20.2	20.8
sigma	1.84	11.1
mn/mx	18/24	19/22.5
<b>1 Week</b>		
avg.	17.0	16.0
sigma	1.78	1.89
mn/mx	15.5/20	14/20
<b>2 Weeks</b>		
avg.	17.9	17.7
sigma	1.63	1.96
mn/mx	15.5/20	14/21.5
<b>4 Weeks</b>		
avg.	18.5	16.9
sigma	1.37	1.70
mn/mx	16.5/20	14.5/19

Claims

1. A plastic closure comprising

a base wall having an inner surface,  
a peripheral skirt extending from said base wall, and

a liner compression molded on said inner surface of said base wall,  
said liner comprising

- (a) ethylene vinyl acetate composition,  
(b) a primary fatty acid amide selected from the group consisting of an oleamide and an erucamide,  
(c) a bis-fatty acid amide,

the total amount of primary fatty acid amide and bis-fatty acid amide being 0.5%-1.5% by weight based on the weight of the ethylene vinyl acetate,  
the ethylene vinyl acetate comprising 85%-94% by weight of polyethylene and 6%-15% by weight of vinyl acetate,  
the ratio of primary fatty acid amide to bis-fatty acid amide ranges between 25:75 and 75:25, by weight.

2. The closure set forth in claim 1 wherein said bis-fatty acid amide is selected from the group consisting of ethylene-bis-oleamide and ethylene-bis-stearamide.

#### Patentansprüche

1. Kunststoffverschluß mit folgenden Merkmalen:

eine Basiswandung mit innerer Oberfläche;  
eine sich von der Basiswandung erstreckende Umfangsringwand; und  
ein auf der Innenoberfläche der Basiswandung formgepreßter Belag folgender Zusammensetzung:

- a) eine Ethylenvinylacetat-Verbindung,  
b) ein Primärfettsäureamid, das aus der Gruppe mit einem Oleoamid und einem Erucamid ausgewählt ist,  
c) ein bis-Fettsäureamid,

der Gesamtbetrag des primären Fettsäureamids und des bis-Fettsäureamids beträgt 0,5 bis 1,5 Gew.%, bezogen auf das Gewicht des Ethylenvinylacetats;  
das Ethylenvinylacetat umfaßt 85 bis 94 Gew.% Polyethylen und 6 bis 15 Gew.% Vinylacetat;  
das Verhältnis des primären Fettsäureamids zu dem bis-Fettsäureamid liegt zwischen 25 : 75 und 75 : 25 Gewichtsanteile.

2. Verschluß nach Anspruch 1, worin das bis-Fettsäureamid aus der Gruppe, bestehend aus Ethylen-bis-oleamid und Ethylen-bis-stearamid ausgewählt ist.

#### Revendications

1. Moyen de fermeture en matière plastique comprenant :

- une paroi de base présentant une surface intérieure,
- une jupe périphérique qui part de ladite paroi de base, et
- une chemise moulée par compression sur ladite surface intérieure de ladite paroi de base,

ladite chemise comprenant :

- a) une composition d'éthylène/acétate de vinyle,  
b) un amide primaire d'acide gras choisi dans l'ensemble formé par l'oléamide et l'érucamide,  
c) un bis-amide d'acide gras,

la quantité totale d'amide primaire d'acide gras et de bis-amide d'acide gras valant de 0,5 à 1,5 % en poids, par rapport au poids de l'éthylène/ acétate de vinyle,  
l'éthylène/acétate de vinyle comprenant de 85 à 94% en poids de polyéthylène et de 6 à 15% en poids d'acétate de vinyle,  
la proportion pondérale de l'amide primaire d'acide gras au bis-amide d'acide gras valant de 25/75 à 75/25.

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2. Moyen de fermeture selon la revendication 1, dans lequel ledit bis-amide d'acide gras est choisi dans l'ensemble constitué par l'éthylène-bis-oléamide et l'éthylène-bis-stéaramide.

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